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OPERATING MANUAL FEED PURIFICATION SYSTEM FOR K-27 3 PP9 11, 12, 14, 15,
FIG III. 1-4, 28-32, 42, 46, 47, 53, 54, 57, 58, 67, 68, 74, 75, 76-80
81-105.

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**EXTRACT OF OPERATIONS MANUAL OM-58
DATED 1/19/46
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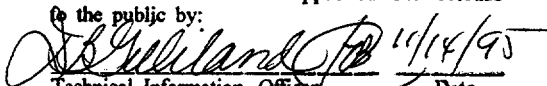
**Compiled by
S. G. Thornton
Environmental Management Division
OAK RIDGE K-25 SITE
for the Health Studies Agreement**

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**Oak Ridge K-25 Site
Oak Ridge, Tennessee 37831-7314
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Technical Information Officer
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This document consists of 108 pages,
No. 15 of 34 copies, Series A, and
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OPERATING MANUAL

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January 19, 1946

INVENTORY

RECORDS DEPT.
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Compiled and Edited by
KELLEX OPERATING DEPARTMENT

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INVENTORIED

OCT 23 1951

By [Signature]

R. F. Benenati
R. F. Benenati

For The Kellogg Corp. M. Benedict
M. Benedict

H. E. Frachtman
H. E. Frachtman

For Carbide & Carbon
Chemicals Corp. C. W. Rucker, Jr.
C. W. Rucker, Jr.

bulb of the Moore Electric Level Controller, a solenoid valve in the drain line is automatically closed, preventing the lighter water layer from entering the drums. This layer is then pumped out of the separator to precipitating tank F-142 by separator pump J-136. This aqueous layer may also be recycled to the extractor for use, in extracting subsequent batches of still bottoms. The above is illustrated in Figure II.1-2.

(c) 616 Absorption

Since Section 130 does not need any cold trap recovery equipment and since carbon traps are not well suited for some of the services required in this section, a water absorption system for removal of 616 is provided. This is illustrated in Figure II.1-2.

Any 616 vapors coming overhead from E-131, from vent gases, from the 616 evacuation pumps, or from relief valve "blows" are fed to the bottom of spray towers E-132A and B. These towers are normally operated in series, but they may be operated in parallel or individually.

A constant bleed of dry air is maintained on the feed line to the tower to prevent back diffusion of water vapor into the system. Water enters the top of the spray towers through a set of spray nozzles, drops through a set of spray nozzles, drops through the tower and drains into one of the circulating tanks F-141A/B. The water in the tanks is then pumped back to the towers by one of the circulating pumps J-133A and B. The gases coming overhead from spray tower E-132A are either passed into the bottom of spray tower E-132B or vented to the atmosphere. The overhead gases from E-132B are vented to the atmosphere. The towers, tanks and pumps are located in a separate building, K-132.

When the concentration of "T" in the water in one of the circulating tanks is built up to the desired level of 1.26 pounds of 616 per gallon of water, the other circulating pump and tank are put on stream and the solution of TO_2F_2 from the first tank is pumped out of the circulating system to the precipitating tank F-142, in K-131. This precipitating tank must previously have been loaded with 60 gallons of 10% caustic solution at 203°F for every 100 pounds of 616 in the solution to be precipitated. The 10% caustic solution is prepared in caustic make-up tank F-143. Air is blown through the caustic solution to provide agitation, and the fluoride solution from the circulating system is run in. Open steam is blown through the mixture as required to maintain a temperature of 203°F during the precipitation. After the solution from the circulating system has been added to the caustic solution already in the precipitating tank, further 10% caustic solution is added slowly to bring the pH of the solution to between 10.0 and 10.5, at which point precipitation of "T" should be complete. The slurry is further agitated by circulating through slurry pump J-134, while adding the caustic. The precipitate, called C-100, contains sodium and oxygen as well as "T".

When the precipitation is complete, the slurry is pumped through filter press G-133. The cake is then washed free of fluoride with warm water, which may be made up in the precipitating tank. The filter cake is dumped from the press and charged to barrels for storage. The filtrate, containing sodium fluoride, is carried to Building K-1407 by tank trucks where the fluoride is precipitated with lime.

column. The E-131 still design was based on a 616:-816 equilibrium diagram assuming that these are the only components to be handled. Five equilibrium trays are sufficient to perform the desired separation. However, twenty-two equilibrium trays are provided to permit the use of this tower to reduce the HF or MoF₆ content of 616 in case this was later found necessary for off-specification feed. E-131 is similar to the stripping tower E-101 of Section 100 in size and capacity; e.g., it will reduce the HF content of 616 from 0.03 to 0.003 wt% and the MoF₆ content from 0.01 to 0.001 wt% at the average rate of 2650 pounds per day.

It was assumed that any decrease in the sharpness of separation of 816 from 616 caused by the increase in column hold-up could be corrected by taking an extra overhead out at the end of a run and returning it to the still with the next batch of waste to be purified. A design H.E.T.P. of 12 inches was assumed for tower E-131 which is consistent with the assumption made in Section 100 design.

In the design of the 816 extraction system, it was assumed that one contact of the contaminated 816 with an equal weight of water as a 2% solution of potassium carbonate would completely remove the 816 present.

(c) 616 Absorption

The spray towers E-132A and B have been designed to handle a maximum feed rate of 1.4 pounds of 616 vapor per second for a total uninterrupted flow of 7-1/2 minutes. The design capacity is the maximum flow that would be obtained if two relief valves in Section 130 "blew" at the same time. Since the design was based on the rate of heat release from the absorption of 616 in water and the maximum allowable temperature rise, without allowing for any transfer of heat from the circulating system to the atmosphere, the absorption system has a capacity appreciably in excess of the design value. The recovery of "T" in the absorption system is expected to be well over 99% of the 616 charged to the system.

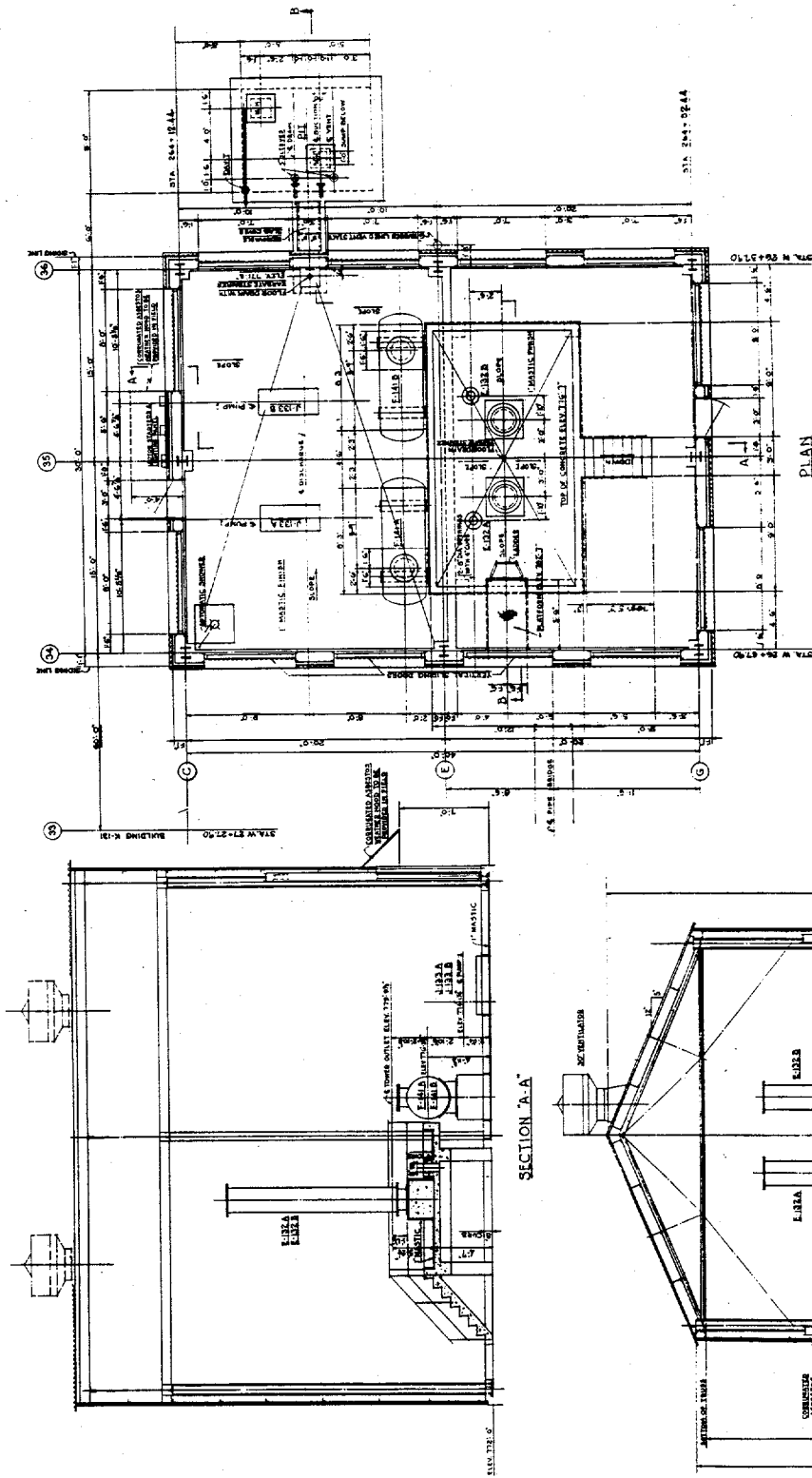
In the design of the spray towers it was assumed that the limiting vapor velocity was that at which entrainment of liquid starts in the gases going out through the vent. A correlation used for calculating this limiting velocity in the case of a plate tower was assumed to give a conservative approximation of the limiting velocity for a spray tower. The design velocity used in these calculations was then taken as 50% of the value calculated from the correlation mentioned above.

In order to calculate the required tower height, a value for the absorption coefficient K_{ga} was assumed based on some data on spray tower absorption found in the literature. A value of 0.25 pound mols absorbed per hour-cubic foot atmosphere was estimated as a fairly conservative coefficient.

In setting the circulation rate in the absorption system, it was assumed that the maximum allowable temperature in the system was 165°F. It was assumed that the off-gas stream comes to the tower at 165°F, and the vent gases and enriched solution both leave the tower at the entrance temperature. On this basis, the only means available for picking up the heat released by the reaction of 616 and water is the temperature rise of the water being circulated through the system, from a normal value of 90°F to a maximum of 165°F. The heat of reaction for the hydrolysis used in the calculations was 111,500 BTU per pound mol of 616 absorbed.

The "T" recovery equipment for this unit was designed to handle a batch consisting of 500 gallons of water containing 630 pounds of absorbed 616. This is the total weight of 616 released in a 7-1/2 minute "Blow" at the rate of 1.4 pounds of 616 per second, absorbed in the total volume of water in the circulating system. In the filter press calculations, it was assumed that a filter cake containing 55% solids on a dry basis would be obtained, that this cake will have a bulk density of 100 pounds per cubic foot and that the cake obtained will be 1 inch thick.

NO. 130 W. 101-D-A



Provision has been made to measure the level, temperature and to measure and control the pressure in the transfer drum F-135. In addition the 74 supply pressure (used in blowing inventory from the drum) is measured.

DUMPING DRUM
F-135 INSTRU-
MENTATION

The level is detected by a 72" internal float Moore transmitter with the float starting 1" from the bottom of the drum. The output of the transmitter actuates PI-236 on the main control board and PI-384 on auxiliary board "C" located at column H-27, first floor. Both gages give the level in % of the float length above the bottom of the float. In general the level is maintained at a height greater than 3".

TE-232 is an iron-constantan thermocouple inserted in a well in the drum and connected to TI-238 on the main instrument board. The latter instrument gives the temperature directly in °F (0-400).

A Moore 1:3 transmitter (0-105 input) is used to measure the pressure in the drum. This actuates PI-234 on the main instrument board and PIC-321 on auxiliary board "C" on the first floor. Both units read directly in psig.

Except when the inventory is under pressure transfer, PIC-321 is used as a relief controller and actuates CV-321. The drum pressure is maintained at 20 psig, except as noted above, by the snap acting direct pilot controller and the spring closed valve. As soon as the pressure exceeds 20 psig, the controller, set on high sensitivity, fully opens the control valve venting the excess inventory. As soon as the pressure drops below 20 psig, the valve is closed.

When the unit is being used to transfer inventory, the control valve diaphragm pressure is bled off by moving the set point of the controller completely off the scale (high side) and by then shutting off the air supply to the controller. The unit may be returned onstream by restoring the air pressure and gradually restoring the set point to the normal control value of 20 psig.

The 74 supply used during transfer operation is given directly by PI-358 located near the casing of F-133B on the floor above the drum. The range is 0-200 psig.

(h) 616 Evacuation System

The 616 evacuation system is used for purging process equipment of 616. The system consists of Beach-Russ Pumps J-132A/B with their associated mist filters, carbon trap F-140, and the associated piping and instrumentation. One of the pumps is a standby spare. The 616 discharged from the pumps is piped to the spray towers for disposal.

DESCRIPTION

Carbon trap F-140 is provided for use in case of emergency. The two pumps and the trap, together with the 216 evacuation pump, are located in the pump room in the northeast part of the first floor. They should not be used to evacuate 216 or moist air.

Hot water must be used in the cooling of the pumps, as cold water may cause condensation of 616. A small shell and tube water heater, C-134 located in the basement near column 30F, is provided for this service. It is capable of providing 250 GPH of 190°F water, using 0 psig steam and 85°F water at 30 psig. A self-contained temperature controller, XX-355, maintains the water outlet temperature by controlling the steam input. PUMP COOLING
WATER

The manual "Installation, Operation, and Maintenance Instructions for Beach-Russ Series 6 Mechanical Vacuum Pumps for Pumping C-616" can be consulted for detailed information concerning operation of the pumps. CARE OF PUMPS
AND TRAPS

The carbon trap is charged, using the usual technique, with a carbon-alumina mixture. The conical portion is charged with 82 lbs. of pure alumina, while the cylindrical portion uses a mixture of 100 pounds carbon with 600 pounds alumina. The alumina is the Norton Company's type 38, 2-4 mesh. The carbon is the high activity, 3/8" pellet, National Carbon Company product.

The 616 evacuation piping, designated "EV" in the piping diagrams and flow sheets, is used to evacuate process equipment of 616. It performs this with the aid of purge headers, designated PU. These joint the process vessels with the "EV" piping, and also with the 74 supply system. This arrangement saves making extra connections on the drums. The description below traces the evacuation piping from the evacuation pumps, J-132A/B, back to the various PU lines. 616 EVACUATION
PIPING

The pump suction lines, EV-51A/B, pass through solenoid valves, as shown in Figures III.2-18 and III.2-19. They join forming EV-52. This receives EV-53, the connection between the pump suction and discharge lines, and becomes EV-1, a 3" pipe which travels west in the casing 16' above the first floor and 8' above the pump room pipe gallery. EV-1 continues west and turns south near column 29F, turning west again at column 29E. At this point continuing west as shown in Figure III.2-4, the EV-1 rises vertically near column 28H. At this point it is joined by EV-5 coming up from the basement, EV-11 coming from the fresh feed vaporizers, and EV-4 coming from the north. This is shown in Figures III.2-10 and III.2-9. EV-5, a 3" pipe, joins PU-31 and EV-16 is the basement, shown in Figure III.2-6. EV-16 is the 2" evacuation line from the waste furnaces. It is valved near column line J. The junction of EV-5 and PU-31, both 3" lines, is valved near column line H. EV-4 joins PU-18 4' north of its junction with EV-1. It is valved at this point. EV-11 is valved near column line 29. PU-31 in the basement serves F-130 through PU-44, F-133B through PU-36B, F-133A through PU-36A, F-135 through PU-38, F-134 through PU-44 and PU-41, and F-136 through PU-44 and PU-33. This is shown in Figures III.2-6 and III.2-5. PU-18 on the first floor serves F-132A through PU-21A, F-132B through PU-21B, F-132C through PU-21C, F-133A through PU-25A, F-133B through PU-25B, F-131A through PU-19A, F-131B through PU-19B, and F-137 through PU-24. This is illustrated in Figure III.2-10.

Underneath the third floor the riser EV-1 joins EV-2 and EV-3, all of which are 3" pipes.

EV-3 is valved into PU-10 3' north of the junction as shown in Figure III.2-12. EV-2 is the line from the reflux drum. It passes east to the vessel and is valved into PU-2, between columns 29H and 30H, as shown in Figure III.2-16. PU-10 serves the rundown drums through PU-13A/B/C, the tower feed drums through PU-11A/B, and the reflux hold-up drum through PU-14, as illustrated in Figure III.2-12. PU-2 serves the reflux drum through PU-3, and the tower vapor line relief valve through PU-1, the reflux drum normal exit line through PU-9, the condenser outlet through PU-4, and the tower vapor line through PU-6. This is shown in Figure III.2-16.

The vessels on the first and second floors are served by purge headers from both above and below. The lower one is generally used in sampling and the upper one for evacuation. The lower purge connection has a tie-in to the upper one, so that it may be used for both purposes. The upper one is not as flexible.

The discharge pipes of the 616 pumps, RV-77A/B are connected with the F-140 carbon trap through RV-78 and the spray tower absorption system through RV-76, as shown in Figures III.2-18 and III.2-19.

A solenoid valve is provided in the common discharge line to prevent water from diffusing into the pumps in case F-140 is substituted for a damaged spray tower. EV-53 allows both the tower feed line RV-86 and the carbon trap line RV-78 to be purged with 74. Although it also allows the relief lines to be used as evacuation lines in connection with F-140, this is not recommended, as water may find its way into the pumps through RV-86, even if it is thoroughly purged and valved off at the towers.

The following variables are measured in the 616 Evacuation System:

616 EVACUA-
TION SYSTEM
INSTRUMENTA-
TION

- (1) Pressure in the header used in evacuation of F-134/135/136.
- (2) Pressure in the headers used in evacuation of
 - (a) F-133A/B
 - (b) F-131A/B and F-132A/B/C
 - (c) F-138
- (3) Main evacuation header 616 concentration.
- (4) Suction pressure of J-132A/B.
- (5) Carbon trap F-140 effluent 616 concentration.
- (6) Circulating water to J-132A/B inlet and outlet temperatures.

The pressure in the purge header PU-31 (see III.2-j below) for drums F-134/135/136 is measured by a C 45 psia Moore transmitter PBM-325 which actuates PI-326/327/328. The latter gages are equipped with a dial that indicates the pressure directly in psia and are located at auxiliary instrument boards "A", "B", and "C" respectively. (Boards "A" and "B" are on basement near drums F-134/136, "C" on main floor immediately above drum F-135).

The pressure in lines PU-27/15/3 are measured by PI-409, 410 and 411 all located on the third floor (elevation 824'-8") at columns H-28 or H-29. These gages have a range of 30" Hg vacuum to 30 psig and can be used during purging as well as evacuation cycles.

The 616 concentration in the main 616 evacuation header is measured semi-quantitatively by XX-511. This unit is a Mark 328 paper type trace indicator which depends upon the collection of 616 or TO_2F_2 on the paper. The paper is treated at definite intervals with a solution of potassium ferrocyanide and a semi-quantitative analysis is obtained by noting the intensity of the brown discoloration. The minimum detectable amount is approximately .011 -.012 mg and quantities of .025 and .035 mg can be easily distinguished from .011 mg. No differentiation can be made with quantities greater than .035 mg.

EVACUATION
HEADER 616
CONCENTRA-
TION

The suction pressures of evacuation pumps J-132A/B are measured by PI-412 and 413 respectively. These gages are mounted near the pumps and have a range of 30" Hg. vacuum to 30 psig.

The 616 concentration in the effluent of carbon trap F-140 is measured semi-quantitatively by XX-441, a Mark 369 crystal type trace indicator shown in Figure III.5-7. This unit absorbs 616 (but not TO_2F_2) in salicylic acid given a bright green to an olive drab coloration depending upon the concentration of the sample stream. A chemical trap is included downstream of the Reagent tube to absorb excess 616 and all of the incident TO_2F_2 and HF. (Spent chemical trap is indicated by a change in color from red to blue).

F-140 616
CONCENTRA-
TION

With a flow of 6 cc/min the discoloration in the Reagent tube moves at a rate of .04 cm/day with a 616 concentration of .1 mg/std liter; with the same flow rate and a concentration of 5 mg/std. liter (spent trap) the rate is 1.3 cm/hr. From the flow rate and the discoloration movement, the 616 concentration can be estimated.

The flow rate is measured by the U tube manometer and regulated by the Moore flow controller included in the apparatus. The flow can be derived by use of the curve in Figure III.5-5 by multiplication by point 1.1.

In operation, the operator should measure the discoloration height and mark this on the provided chart. A maximum of from 1/2 to 1 hour should elapse between readings when the trap is in use.

In putting the unit onstream the outlet valve must be opened before the downstream valve, in taking the unit offstream the downstream valve must be closed first, or the oil from the manometer may be blown into the system.

At the time the Reagent tube is replaced the adsorbent tube should also be replaced to ensure optimum operating conditions for the unit.

Five 20° - 240°F indicating thermometers measure the pump water temperatures. TI-401 is at the exit of C-134, TI-534 at the inlet to J-132A, TI-535 at its outlet, TI-536 is at the inlet to J-132B and TI-537 at its outlet.

(i) Relief Line Piping

The relief piping, designated RV, carries 616 from relief valve blows, 616 from the J-132 pumps, and light impurities from E-131 to the spray towers E-132A/B for absorption in water.

The description below follows the RV piping back from the spray towers. Figure III.2-18 shows RV-86, the 4" pipe running from the rubber lined pipe in K-132 over the pipe bridge to the K-131 vacuum pump room. In K-132 a continuous bleed of dry air is maintained where RV-86 joins the rubber pipe. This prevents back diffusion of water vapor, which will be very corrosive. RV-86 is not encased, but is electrically heated to prevent condensation of both 616 and water vapor. From the pump room, RV-1 travels in the same casing as EV-1 to column 29H, as shown in Figure III.2-11. It continues westward and receives RV-39 from the basement, and RV-20 from the north, as shown in Figures III.2-6, III.2-9 and III.2-10. RV-39 is the result of the union of RV-51 and RV-24 in the basement near column 28H. The former comes from the waste furnaces, being valved (locked-open) just outside the furnace room. The latter is valved (locked-open) near the junction and furnishes F-135 through RV-30, F-134 through RV-38 and F-136 through RV-26 and is shown in Figures III.2-6 and III.2-5. RV-20 is valved (locked-open) 4' from the junction with RV-1. It takes care of F-133A through RV-22A and F-133B through RV-22B, as shown in Figure III.2-10.

From this junction RV-1 ascends, receiving RV-2 and RV-11 under the third floor, as shown in Figures III.2-12 and III.2-9. Both of these are 3" pipes. RV-11 is valved (locked-open) and supplies F-131A, F-131B, F-137, F-132A, F-132B, F-132C respectively by means of RV-13A, RV-13B, RV-19, RV-17A, RV-17B and RV-17C, as shown in Figure III.2-12. RV-2 comes in from the east, paralleling EV-2 as shown in Figures III.2-13 and III.2-16. It is valved (locked-open) between columns 29H and 30H. It takes light material from the tower through RV-4, excess 616 from F-138 through RV-3, and excess 616 from the tower vapor line through RV-5.

4. 616 Absorption System

(a) Gas Piping

The 616 absorption system is located in Building K-132 and is illustrated in Figures III.4-1 and III.4-2. 616 is brought over the pipe bridge by means of RV-86, a four inch nickel plated pipe, not illustrated in Figure III.4-1. In the southwest corner of the room it joins the rubber lined piping. As RV-86, it enters the base of E-132A. Continuing east as RV-87, it enters E-132B. A pipe from the top of E-132A comes down between the towers, also designated RV-87 and joins the horizontal pipe. These pipes, all of four inch size, are valved so that the gas may flow through the towers in series or in parallel. This is illustrated in Sections "H-H", "G-G" of III.4-2 and "Plan at AA" of III.4-1. The scrubbed gas leaves the towers through RV-88 and RV-99 and passes out through the vent stack on the east side of the building. This is shown in Section "F-F" and "Plan at CC".

A dry air bleed is made into RV-86 using DA 21. DA 130-58 is the vent line from the conditioned casings.

(b) Liquid Piping

The aqueous solution leaves the circulating tanks through AC-1A and B, shown on Figure III.4-1, and enters the pumps to the north. AC-2 allows either pump to be used with either tank. AC-3A and AC-3B are discharge recycle lines, and join AC-1A and B. Also, the three inch pipe from the sump joins the pump suction lines as shown in Section "D-D". Section "EE" shows the discharge lines. Material may either be pumped through AC-12 to F-142 in K-131, shown in Figure III.5-1, or it may be pumped to the towers through AC-4. This 3" pipe passes over and between the circulating tanks and enters the towers through the nozzle system. This is shown in Sections "H-H", "E-E" and "F-F" and "Plan at BB".

Liquid from the towers flows out through traps into the circulating tanks, as illustrated in Sections "F-F" and "H-H" and "Plan at AA". Provision for over-flow is made by means of open funnels and the "S" shaped traps shown in Section "F-F". The extra liquid is drained to the sump outside the east wall, from which it can be recovered by using the J-133 pumps.

AC-9A and B are the three inch pipes connecting the towers and tanks, and AC-10 is the cross-tie. A 1-1/2" pipe, AC-11, vents both tanks to the vent stack.

(c) Sump Piping

There are four pipes leading into and out of the enclosed

5. C-100 and 816 Recovery System

(a) C-100 Recovery System

The equipment in the C-100 Recovery System is made up of the following:

- (1) A rubber and carbon brick lined precipitator F-142 for precipitating "T" salts from the enriched solution.
- (2) A steam jacketed caustic make-up tank F-143 for making up caustic solution to be used in precipitation.
- (3) A plate and frame filter press G-133 for separating precipitate from the mother liquor.
- (4) A diaphragm type slurry pump J-134 for circulating slurry and feeding the filter press. A simplified engineering flowsheet of the recovery system is given in Figure V.2-4. The piping drawings are given in Figures III.5-1, III.5-2 and III.5-3.

The caustic solution which is used to precipitate the 616 is prepared in tank F-143, located on a special platform in the northeast corner of the first floor.

CAUSTIC
MAKE-UP

The 50% caustic solution is added to the vessel through 1-1/2" line CS-1 and the diluting water is taken from a 1-1/2" line from the sanitary water system. The temperature of the caustic solution is maintained at the desired value by either adding steam to the vessel jacket through 1-1/2" S-27 or by adding cooling water through 1" CW-26. The vessel jacket is provided with a 1" drain line equipped with a steam trap and a 1" relief valve set at 20 psi.

The caustic make-up tank is also provided with a 55# dry air line which is equipped with a flow element and a differential indicator. By allowing only a few bubbles of air to pass the flow element and observing the pressure on the differential indicator, the height of liquid in the tank can be determined.

Before pumping the acid solution from K-132 to the precipitator F-142, the precipitator is charged with 62 gallons of 10% caustic solution for every 100 lbs of 616 in the acid solution to be precipitated. The caustic is drained to F-142 from the caustic make-up tank by means of 2" line CS-2. Air is blown into the tank through 1" line A-11 to provide agitation and the acid solution is pumped to the tank from Building K-132 by means of 3" line AC-12.

616 PRECIPITATION

Open steam is added through 2" line S-28 as required to maintain the temperature at 203°F during precipitation. The slurry may be further agitated by circulating through AC-13, the slurry pump, AC-16, and back into the tank through AC-15. The

precipitating tank F-142 is provided with an air bubble liquid level indicator similar to that on F-143.

When precipitation is complete, the slurry is pumped by means of a 3" lines AC-13 and AC-14 and the slurry pump J-134 through the plate and frame filter press G-133. The filtrate may then be recirculated to the tank through 3" line AC-15 or it may be discharged directly to tank trucks which will transport the liquor to Building K-1407. Warm water is then prepared in F-142 by adding open steam to process water. The filter cake is then washed with this warm water and the washings added to the filtrate in the tank trucks. Finally the cake is dumped to a bin below the press. From here it is charged to barrels for storage.

FILTRATION

The steam pressure supplied to the jacket of F-143 is indicated on PI-291, a locally mounted gage with a range of 0-60 psig.

INSTRUMENTATION

The liquid level in this drum is measured by an air bubbler level indicator DI-423. This unit has a range of 0-87 inches of a liquid of density 1. The dial is calibrated in terms of % of the total range which is designed to start at the bottom tangent line. The air supply for the bubbler is controlled to a value of 4 psig by PCV-426. The unit which restricts the flow is FE-424, similar to FE-287 described previously.

The precipitator F-142 is provided with the following instrument services.

Precipitator temperature - The temperature of the solution in tank F-142 is indicated by TI-250, a vapor pressure actuated dial thermometer with a range of 50-240°F.

Steam supply pressure - This is indicated on PI-249, a locally mounted pressure gage with a range of 0-30 psig.

Agitation air supply pressure - This is indicated on PI-290, a locally mounted pressure gage with a range of 0-160 psig.

Drum level - The level in drum F-142 is measured in a similar manner to F-143. The air pressure is controlled by PCV-426; the air flow is restricted by FE-422, and the level is read on indicator DI-421.

The discharge pressure of the slurry pump J-134 is given on PI-251 (range 0-100 psig). This gage is mounted right above the pump and is protected by a water purge stream. Since the discharge pressure of the pump varies and may rise as high as 100 psig, it is necessary to provide a water purge stream at 100 psig. This is done by filling two sections of 12" schedule 40 steel pipe with water and then opening the "pipe tanks" to the full pressure of the air lines which is 100 psig. The tanks

Near this valve GE-1 extends down to the basement, shown in Figure III.7-5. The three vessels located here, F-134, F-135 and F-136 are supplied as shown. GE-9, the three inch pipe which feeds GE-1, is valved along column line "H" as shown.

EASEMENT

GE-3 joins GE-1 underneath the second floor as shown in Figure III.7-4. Coming down from above, it provides for F-131-A and B, F-132-A, B and C, and F-137 as shown in Figure III.7-6. GE-3 is valved along column line "H". A two inch branch of GE-3, GE-2 takes care of F-138, as shown on Figures III.7-6, III.2-16 and III.2-17.

SECOND FLOOR

216 can only be admitted to this system through the single flange mentioned above. The GE piping does not enter a vessel directly, it always connects with the PU piping, except in the case of F-138, where it connects jointly with RV-5 and F-132-A, B and C, where it makes use of the PL piping.

The instrumentation of this system is listed on Table III.2-1 and illustrated in Figure III.2-22.

(c) Moisture Purging System

The moisture purge system is used for purging the breakable 616 cylinder connection of wet air before exposing them to 616. It consists principally of carbon traps F-448-A and B connected to the suction pipes of Beach Russ pumps J-138-A and B, together with the necessary instrumentation and piping, designated WE and illustrated in Figure III.7-7. The pumps are the standard commercial type, using hydrocarbon lubricating oil. The carbon traps are used only to pick up traces of 616 which may come from leaking valves, and therefore 216 or 616 must never be evacuated through this system to avoid causing considerable damage. One pump and trap are standby spares.

The two pumps and traps are located in the northwest corner of the shipping drum feed room. PI-444, a locally mounted 30"-0-30# gauge, measures the upstream pressure at both traps. Similar gauges PI-445 and PI-446 measure the pump suction pressures. Trace indicators XX-442 and XX-443 mounted respectively on the inlet and outlets of the carbon traps inform the operator of the possible presence of 616 in the purged gases and of the eventual depletion of carbon charge.

The traps are each loaded with 38 pounds of 3/8" pellets of high activity carbon (from the National Carbon Company). No alumina is used, as the 616 content of the gases should be very low.

The instrumentation of this system is listed in Table III.2-1 and illustrated in Figure III.5-4.

(d) Hot Water System

The hot water circulating system, which provides hot water to melt 616 in the waste furnaces and vaporize it in the feed vaporizing baths, consists of the following pieces of equipment, together with the necessary piping and instrumentation; one circulating tank F-147, two pumps, J-135-A and B, four hot water baths, F-146-A, B, C and D and three waste furnaces B-131-A, B and C. The piping diagrams for this system are given in Figures III.7-8 and III.7-9.

DESCRIPTION

The water tank, F-147, has a total capacity of 3450 gallons, and when filled to a level of 4'-6" (2800 gals.) it should contain enough water to fill all four baths and still have the steam heating coil covered, which requires a 20" level.

The circulating pumps, J-135-A and B, are controlled by locally mounted push buttons. Three lights for each pump are provided on the shipping drum room control panel. Green indicates that the pump is normally off, red means on, and amber means that pump is off due to trouble.

The instrumentation at the hot water tank F-147 and the two circulating pumps J-135-A and B is illustrated in Figure III.2-22 and is given in detail on Table III.7-2. PI-112 and PI-113, 0-60 psig locally mounted gauges, measure the pump discharge pressures. Gauge glass GG-110 on F-147 serves to indicate the amount of water in the drum. The steam inlet to the drum is provided with CV-311, a spring closed diaphragm control valve. It is operated by TIC-111, an inverse pilot bulb type temperature controller, set to maintain the drum water temperature, at 200°F. Two temperature switches TBS-390 and 393, are installed in F-147, to be used as high and low temperature indicators. The former operates alarm lights AI-391 and 392 and the latter AI-394 and 395. The first of each set are mounted on the central panel of the shipping drum room instrument board. The other two are on the waste room signal board. TBS-390 is the high alarm, set at 205°F, and TBS-393, set at 195°F, is the low temperature alarm.

INSTRUMENTATION

(e) Steam Supply

Figures III.7-10 and III.7-11 illustrate the steam system in Building K-131. The supply pipe comes in over the Section 400 bridge and is valved immediately inside the building at column 28L. The supply pressure of 35 psig is reduced to 15 psig by PCV-336 located in the basement near column 29J. This steam is

DD damper and one for the two outer ones. The remaining five have individual buttons, suitably marked. Pressing the button marked "open" will apply 15 psig air to the appropriate motor, and open the damper completely. Pressing the "closed" button will cut off the air, bleed the motor, and completely close the damper.

The four fans are No. 6-1/2 type "LL" Buffalo Forge units. They are rated at 19,000 cfm, 1-3/4" S.P. at 95°F and 70% R.H. They require 7.86 HP at their speed of 670 rpm. The limit load is 8.81 HP. The 1740 rpm motors turn them by means of Texrope drives on 52.1" centers.

There are two push button stations for each of the four ventilating fans. One set, without lights, is mounted on the east wall of the fan room, along column line 25. The other set, with red and green indicating lights, are on panel 11 of the main control board. The wiring diagram for these stations is given in Figure III.3-3. The red light indicates that the associated fan is operating.

The exhaust component of the ventilating system consists of 17 roof ventilators with 13 automatically controlled dampers.

EXHAUST
VENTILATION
SYSTEM

There are two motor operated ventilators, equipped with self-acting louvers, in the roof of the wastedrum unloading room, and four more in the roof of the shipping drum feed room. All six have Davidson Hyduty V-belt driven ball bearing fans, size #22. They have a capacity of 3160 cfm and revolve at 1150 rpm. The 3/4 HP 440 volt, 60 cycle, 3 phase driving motors turn at 1750 rpm. The push buttons for these fans are located on either side of the south truck entrance vestibule.

The air supply for these rooms is obtained through 9 sets of louvers in the south wall of the building just above ground level. The louvers are manually operated by a removable crank. Behind the louvers are mounted down swinging replaceable filter frames.

There is a 16-3/4" x 3-5/6" Burt Monovent continuous ridge ventilator in the northeast corner of the 4th floor roof. Four ducts open into this ventilator, from the four floor levels directly below. Automatically regulated dampers are mounted over the interior openings of this duct. In the basement, over the coolant drum is damper DL. In the ceiling over the coolant cooler is damper DM. The third and fourth floor ceiling dampers are DN and DO, respectively.

The 10 remaining ventilators are Burt Free Flow units, differing only in size and minor details of construction. A 36" ventilator is mounted in the northeast corner of the first floor roof, at the end of a 36" square duct which comes directly up from damper DQ in the basement ceiling.

Three 20" ventilators are located on the roof of the first floor auxiliary equipment room. No ducts are supplied, dampers DR are supplied integrally mounted.

A 12" ventilator with damper DS is situated over the F-140 carbon trap.

Two 16" ventilators, without ducts but with dampers DV are provided in the roof of the first floor fan room. Two 30" ventilators are mounted in this section also. They are at the outlets of 28" square ducts, without dampers, which extend directly up from the basement transformer room ceiling. The remaining ventilator, 36" in size, is mounted on the roof of the fourth floor penthouse. It is at the top of a 38" x 26" duct which comes up from the tower room second floor ceiling level. DW is the damper at this location. There is also a damper DX at the fourth floor ceiling which communicates with this ventilator.

All 13 of these exhaust dampers are operated by Minneapolis-Honeywell type P-0900-A static pressure regulators which are located in the room associated with the particular damper. The building pressure is maintained at 1/8" W.C. above outside atmospheric pressure.

inlet and outlet, and G is the level indicator flange. D is the purge line nozzle, E is the relief nozzle, and F is the high-pressure 74 inlet. The total capacity is 13,000 lbs 616 at 165°F.

(h) Reflux Hold-up Drum

The reflux hold-up drum, which is located on the second floor next to the three rundown drums and is shown in Figure IV.2-8, is a vertically mounted cylinder 10" ID x 2'-0" tangent to tangent. It is made of 3/16" monel metal, and is supported 2'-5" off the floor by three 2" x 2" x 1/4" angles welded to the shell. Its total capacity of 165°F-616 is 325 lbs.

Nozzle A is the relief, purge, and 216 line outlet. B is the liquid outlet and C is the inlet. D is the thermocouple connection.

(i) Reflux Drum

The reflux drum, F-138, which is located near the top of the distilling tower on the third floor, is illustrated in Figure IV.2-9. This is a vertical cylindrical tank 18" ID x 4'-0" tangent to tangent and is constructed of 3/16" monel metal. The tank is supported 4'-6-1/2" off the floor by three 3" x 3" x 3/8" angles welded to the shell.

Coupling A is a 2" connection which serves the 216 evacuation and 74 lines associated with the drum. Nozzle B is the 2" outlet connection and C is a 1" relief vent connection. E is the 2" liquid inlet and F is a 3/4" coupling for the level indicator. G is a 3/4" thermocouple well and D is a 6" hand hole, H is a 4" Sargol joint to which is attached a 2'-0" length of 4" pipe connections, G and B are in this length of 4" pipe.

(j) Carbon Trap

The 616 carbon trap, F-140, located at the north end of the first floor evacuation pump room, is illustrated in Figure IV.2-10. It is supported on the steel platform by two lugs extending from the wide central section. The 616 entrance pipe is a 6" flanged connection marked A. There is a 1-1/2" exhaust gas outlet B near the top. Two 1/2" 74 inlets, G and H, are provided although only one is to be used. The other is plugged. A dump gate at the bottom is used for emptying and a manhole at the very top enables the trap to be recharged. Only one thermocouple well is provided, marked F. The vessel is made of 3/8" carbon steel.

(k) Moist Air Carbon Traps

The two moist air carbon traps F-148A/B which are located in the northwest corner of the shipping drum feed room are illustrated in Figure IV.2-11. They are vertical cylindrical tanks with a dished

head on top and a flat plate on bottom. The unit stands 3'-1" off the floor and is supported on three angle iron legs. The plate sealing the bottom of the drum is bolted to a flange which is welded to the shell. A special hinged connection is provided so that the bottom plate may be swung out of the way for discharge.

The inlet pipe is a 2" connection marked C in the lower part of the shell and the outlet pipe is a 2" connection marked A in the top dished head. The top head is also provided with a 4" hand hold to be used in charging and discharging. No thermocouples or TC connections are provided.

Emptying is accomplished by unbolting the bottom hinged cover and inserting the special plug in the inlet pipe. The screen is then unbolted and the top flange opened, so that the screen may be lowered by the screen hook and the contents discharged. The screen is then bolted into place and a new charge is added through the top. The special plug is then removed and both covers are closed. The vessel is constructed of 3/8" carbon steel.

(1) Spray Tower Circulating Tanks

The spray tower circulating tanks, F-141A and B, shown in Figure IV.2-12, are horizontal cylindrical tanks 3'-0" ID x 8'-3" tangent to tangent. These tanks, which are located in Building K-132, are constructed of rubber-lined 5/16" carbon steel. The lining is 3/16" thick of Goodrich "Triflex R". Connection A is an 18" manhole, C is a 3" flanged connection for the dry air supply, E is a 3" flanged connection for the inlet from the spray tower, and B is a 1-1/2" vent. D is a 3" flanged connection on the bottom of the tank for the outlet pipe.

(n) Precipitator

Figure IV.2-17 shows the precipitating tank, F-142 which is located in the northeast corner of the basement. This is a vertical cylindrical tank 7'-0" ID x 6'-0" tangent to tangent x 3/8" thick. The vessel stands 3'-11" off the floor and is supported on four 6" x 6" x 3/8" angles welded to the shell. Flange A is a 24" manhole. F and B are 6" flanges for the steam and air agitation pipes. D is a 4" vent connection, and C is a 6" solution outlet. Process liquid enters through G from J-133A and J-133B and through E from J-134. H and J form an inlet for a #21 Carbate thermowell in which is a dial-type vapor filled thermometer.

Although it is not shown in the drawing, the vessel is lined inside with 3/16" of Goodrich "Superflexite F" which will stand 180°F. This lining covers the whole interior, including the outside faces of the flanges. Inside of the rubber lining is another of 4-1/2" thick

5. Miscellaneous

(a) Shipping Drum

The shipping drum illustrated in Figure IV.5-1 have an internal volume of 2.41 cu ft and hold about 450 lbs of 616. One head of the tank is ellipsoidal while the other is spherical. Both heads are equipped with Kerotest 440-A3 Chlorine Institute valves and 3/16" metal skirts. The valve in the spherical head is equipped with an internal ram's horn pipe.

(b) Liquid Waste Drums

The liquid waste drums are modified standard one ton liquid chlorine cylinder. The modification consists in removing one of the two internal ram's horn with its 3/4" valve. The valve has been placed in one of the three fusible plugs on the same end of the cylinder. The other plugs have been removed and the connections have been plugged and sealed. All of the liquid contents is removed through the ram's horn, which must be positioned in a vertical plane. The other valve is for venting. These cylinders have been obtained as surplus property from the Chemical Warfare Service. They weigh 1300 lbs empty, have a capacity of 25.76 cu ft, and may hold between 4000 and 5000 lbs of waste.

(c) Waste Furnaces

The three waste furnaces B-131A, B and C are illustrated in Figure IV.5-2. They are located in the liquid waste unloading room on the west side of the south truck entry vestibule. The liquid waste cylinder, having a volumetric capacity of 25.76 cu ft, are placed in them horizontally and the liquid contents are siphoned out through the ram's horn pipe inside the cylinder. Hot water from F-147 is circulated through both the upper and lower pipe coils to liquify the cylinder contents. The coils of the bottom half are permanently connected to the hot water piping while those of the removable top half connect with it through flexible hoses. Not shown in the drawing, but part of the top half outlet piping, is an inverted "U" shaped pipe, by-passed by a valve and extending over the top of the furnace. With the valve closed, it serves to trap water in the coils of the top outlet half, as with low pumping rates this portion may drain too rapidly.

(d) Filters

The filter press G-133, located in the northern part of the auxiliary process room in the basement, is a standard piece of equipment, a product of T. Shriver and Co. It contains twenty-two 24" sq chambers. The frames are 1-1/2" thick. The four wash and feed supply and discharge pipes, located in the four corners, are 1-1/2" in diameter.

PLATE &
FRAME PRESS

The still bottoms filter, G-131, illustrated in Figure IV-5-3 is a horizontal cylinder 6" ID x 2'-0" long the outlet nozzle is marked A, the inlet E and the drain nozzle C. The entire inside is filled with nickel wool and the outside is coated with 1" of lead, to afford protection against the radioactive products which will accumulate in the filter. The unit is mounted horizontally and is constructed of 3/16" monel metal.

STILL
BOTTOMS FILTER

V. OPERATING INSTRUCTIONS

1. Fresh Feed Vaporization System

Fresh feed is received at Section 130 in 150 lb. water capacity cylinders, equipped with a valve and internal ram's horn pipe at one end and with the valve only at other. The cylinders have been filled at 212°F by the supplier to approximately 90% of their capacity with 616, and then pressured with 74 at approximately 68°F, to 5 psig. On this basis, cylinders contain 463 lb. of 616, and have a gross weight of 663 lbs. The internal volume is 2.41 cu. ft.

The presence of 74 in the cylinders will result in cylinder pressures in excess of the normal vapor pressures of 616. The following developed pressure figure, are based on the 2.41 cu. ft. cylinder being filled with 463 lbs. of 616 and then pressured to 5 psig with 74 at 68°F, and on the assumption of zero solubility of 74 in 616.

DRUM CONTENTS TEMPERATURE	STATE OF CONTENTS	CYLINDER PRESSURES		
		WITHOUT INITIAL :REMOVAL OF 74	PARTIAL REMOVAL: : OF 74 (1)	COMPLETE :REMOVAL OF 74
68°F	Solid	: 5 psig	: -17.7 psig	: -13.2 psig
147°F	Solid	: 28 psig	: 8.1 psig	: 7.5 psig
147°F	Liquid	: 58 psig	: 8.7 psig	: 7.5 psig
180°F	Liquid	: 87 psig	: 24.1 psig	: 12.8 psig
212°F	Liquid	: 132 psig	: 47.0 psig	: 45.8 psig

(1) Pressures developed following partial removal of 74 are based on the cylinder being evacuated to 2 psia at 68°F.

The maximum feed rate from Section 130 to Section 400 is 12,600 lbs of 616 per day (normal 6000 to 9300 lbs. per day.)

(a) Drum Handling

Lift cylinders one at a time from truck using overhead hoist and a Mansaver 42" lifting bar. By previously taring the weight of hoist, filter bar, etc., on the tare beam of the Fairbanks overhead track scale, the weight of cylinder and contents can be read directly and recorded by the operator, as the cylinders are moved from the truck to the drum tilting rack in the Shipping Drum Feed Room. The cylinder is then picked up off the tilting rack, using the overhead hoist and a Mansaver 15" lifting bar, and placed vertically in the drum storage rack. In order to avoid rehandling, the cylinder should be grabbed and placed in storage rack so that the end equipped with the internal ram's horn pipe will be vertically down. OPERATION

By weighing the empty cylinders as they are loaded back onto the truck, the 616 input to Section 130 can be determined by weight difference. Empty cylinders can also be stored in the storage rack until trucking facilities are available to remove them.

B. Preparation of Bath for Placing Onstream

The following operations are outlined for application to water bath F-146A, but pertain to all of them:

- (1) Place four cylinders, one at a time, into the drained water bath (F-146A), using the overhead trolley system. The cylinders should be loaded in the bath so that the end equipped with the internal ram's horn pipe is down. Connect the cylinders to the bath manifold using flexible metal hoses.
- (2) With all other manifold valves closed except CV-270 open the manifold valves at the individual cylinders.
- (3) Pressure the bath manifold and the individual cylinder connections with 20 psig 74 using the evacuation line double block valve 74 connection. Check the connections for leaks by inspection and by pressure readings. Pressures may be read on PR-101 (blue pen) and also at each cylinder by local gages. If the drop in pressure is less than 1/4 psi in one-half hour, the connection may be considered tight.
- (4) If all connections are satisfactory, pump out through the moist air evacuation connections, using J-138A or B. The pneumatically operated block valve, as well as the individual cylinder valves should be closed while doing this. The procedure is to evacuate the connections to 0.2 psia, close the evacuation valve and pressure with 74 to at least 15 psig. Evacuate again to 0.2 psia and re-pressure as before. Then evacuate to as low a pressure as possible and close the evacuation valve.
- (5) Close the manifold valves at the cylinder connections and check the individual cylinder pressures by opening the cylinder valves. If a cylinder pressure is not above atmospheric, as indicated by the gages on the individual connections, the cylinder may have been contaminated by wet air in transit or storage. In such a case, the cylinder valve or the cylinder concerned is closed, the connection purged as described below, and the cylinder is removed from the bath to be replaced by a fresh one.

- (6) When all the cylinders have been checked in this fashion, open the manifold valves in the cylinder connections, thereby connecting the cylinders to the manifold. With block valve CV-269 open, remove the 74 from the cylinders, evacuating to at least 2.0 psia pressure. Use the 616 evacuation system with J-132A or B.
- (7) With block valve CV-269 closed and the valves at local gages PI-105, PI-106, etc. closed, fill the bath with hot water and start the water circulating. Water flow should be adjusted to maintain bath temperature of 195° to 200°F. The pressure gages are normally used only to check the cylinder connections and should not be used on 616 operation, if possible.
- (8) When the manifold pressure is up to 20 psig, as indicated by PR-101 (blue pen) and by alarm light AI-267 going out, the bath is ready to be placed onstream.

(c) Placing one bath on stream and taking another one off:

Individual baths should be placed onstream in stepwise fashion, that is, a fresh bath of cylinders should be placed onstream before the bath already onstream is exhausted or empty. Operating experience will indicate how long it takes to completely vaporize a bath of cylinders, and consequently how long a bath may be onstream before adding a fresh bank of cylinders to the main feed header. By placing a fresh bank onstream prior to complete vaporization of the bank onstream, there will be insured a constant feed pressure to the feed filters in Section 400. To place another bath onstream, prepare it as outlined above, and when desired, merely open the SMP and pneumatic block valves.

- (1) When F-146A manifold pressure has dropped to 20 psig as indicated by PR-101 (blue pen) and by red alarm light AI-267 going on, the operator should watch the manifold pressure and when it drops to the main feed header pressure of 15 psig (shown by PRC-102, red pen), he should close block valve CV-269, by turning the three way cock mounted below the recorder.
- (2) With the manifold SMP valve closed and the air block valve open, pressure the cylinders with 74 to 20 psig and evacuate to 0.2 psia. Do this twice more, evacuating to as low a pressure as possible on the third evacuation. Then refill to at least 15 psig with 74 and close the cylinder, manifold and air block valves. Break the 616 cylinder connections.
- (3) Drain the hot water from the bath and remove the cylinders, one at a time, placing them in the cylinder rack.

(d) Procedure for placing system onstream when the vaporizers of the Purification Systems are not onstream:

This condition might occur on initial startup of the K-27 plant; on resumption of operations following shut down of either Section 130 or Section 400; or following interruption of feed to Section 400 resulting from possible yard trouble.

- (1) Condition all piping and equipment that will be exposed to 616 in Building K-131 and the yard.
- (2) Place the electric pipe line and casing heaters in operation to preheat the piping and equipment in Section 130. See that the yard line is also up to operating temperature.
- (3) Place the various services and systems that will be needed in Building K-131 and K-132 in operation. These include:
 - (a) 616 Evacuation System
 - (b) Moisture Purging System
 - (c) Hot Water Circulating System
 - (d) 616 Absorption System
 - (e) Air Distribution System
 - (f) G-74 Distribution System
- (4) Place in operation and bring up to pressure one or more water baths of cylinders so that they are ready to be placed on stream.
- (5) When the Section 400 operator indicates he is ready, the Section 130 operator can place the first bath of cylinders on stream by opening manifold block valve (CV-269 in the case of bath F-146A). The bath SMP block valve should be open, as well as the valves separating PG-12 from PG-11, located in the southwest corner of the shipping drum room. The PG-3 valves at this junction should be closed. Of course the yard line should previously have been evacuated of 74.

(e) Placing System on Stream When the Vaporizers are Onstream:

This condition might occur following completion of a batch distillation run of 616 or following shut down of the batch distillation equipment for other reasons.

- (1) Condition the additional piping and equipment that will handle 616 in Section 130.
- (2) Preheat the additional piping and equipment that will be used.
- (3) With the block valves in line PG-12, at the junction point of lines PG-3, PG-11 and PG-12, still closed, bring up to pressure one or more water baths of cylinders so that they are ready to be placed onstream.
- (4) With the manifold block valves (CV-269 in case of bath F-146A) closed, open the block valve in line PG-12 (see Step "3" above). This will place the Shipping Drum Feed Room main header (PG-12) under pressure of the vaporizers (F-133A or F-133B).
- (5) Continue feeding Section 400 with feed from the evaporators (F-133A or B) in this manner until it is desired to put the Fresh Feed Vaporization System onstream. At this time place the first water bath of cylinders onstream by opening its manifold block valve (CV-269 in case of bath F-146A), having first opened the manifold SLP block valve.
- (6) After placing the Fresh Feed Vaporization System on stream (Step "5" above, close the block valves in Line PG-3 at the junction of lines PG-3, PG-11 and PG-12.
- (7) For additional steps required to complete shutdown of the evaporators (F-133A or B) refer to Section V-2-f.
- (8) Additional baths of cylinders may be placed onstream as needed.

2 616 Purification System

(a) Cylinder Handling

The cylinders are hoisted from the truck, one at a time, using the overhead hoist and a Mansaver 78" lifting bar. In order to avoid rehandling, grab the cylinder with the lifting bar so as to have the internal ram's horn pipe of cylinder vertically down. By previously taring the weight of the hoist, lifting bar, etc., on the tare beam of the Fairbanks scale, the weight of the cylinder and its contents can be read directly and recorded by the operator as the cylinders are moved from the truck to the furnace. Place the cylinder in the furnace so that the cylinder valves are at the open, north end of the furnace and so that the internal ram's horn is vertically down.

With the cylinder in place in the furnace, lift the top half of the furnace from the floor, using the 78" lifting bar, and lower it into position. Tighten the two halves together using hinge bolts. Make the necessary water hose connections.

When the liquid unloading is complete (see next section) and the water hose connections are broken, loosen the furnace hinge bolts and lift off the upper half of furnace, and place it on the floor. Remove the empty cylinder from the furnace. Weigh the empty cylinder as it is moved from furnace to truck, so that the waste input to Section 130 can be determined by weight difference.

(b) Furnace Operation

The following operations are outlined for application to waste furnace B-131A, but pertain to all three of them. Refer to Figure V.2-1.

- (1) With the furnace loaded as described in the previous section and with the cylinder valves closed, connect the cylinders to the piping system using the flexible metal hose. Connect line PL-51 to the centermost cylinder valve (the one equipped with the internal ram's horn), and RV-52 to the outside cylinder valve.
- (2) With the cylinder valves closed and the header valves in both PL-51 and RV-52 closed, pressure lines PL-51 and RV-52 up to the cylinder valves with 20 psig 74 from the evacuation line double block valve 74 connection. By having the valves open in crossover line, RV-53, both cylinder connections can be pressured and checked simultaneously. Check the connections for leaks by inspection and by pressure readings (PI-454). If the pressure drop is less than 2/4 psi in one-half hour, the connections may

be considered tight. If a leak is indicated when testing both connections simultaneously individual connections may be isolated by closing the proper valve in line RV-53

- (3) When the cylinder connections are satisfactory moisture purge the two connections simultaneously. With all furnace and cylinder 616 valves closed except the two in crossover line RV-53 and the RV-52 and PL-51 hose valves, evacuate to 0.2 psia through WE-7 by opening the two valves in this line. Then pressure to 15 psig with 74, evacuate again to 0.2 psia and repressure to 15 psig. Finally, evacuate to as low a pressure as possible and then close the two WE-7 valves
- (4) Open only the outermost cylinder valve (connected to line RV-52) and using the 616 Evacuation System evacuate the 74 from the cylinder down to a pressure of at least 2 psia, and then close the evacuation valve.
- (5) With the valving as in step (4) above, start the hot water flow through furnace. When cylinder pressure readings (PI-454) indicate that the contents are melted, (See the Table on page), close the crossover valve nearest PL-51, and sample the contents. A recommended sampling procedure is as follows:

Connect the previously evacuated receiver to the sampling line. Evacuate the sampling line up to the receiver through EV-17, then open the receiver valve. Close the evacuation valve and pressure the receiver with 74. Evacuate and pressure with 74 again, and finally evacuate to as low a pressure as possible and close the evacuation valve. Open the cylinder center valve and both sampling line valves, allowing the liquid to fill the container. Then close the receiver and the sampling line valve nearest PL-51. Evacuate the pipe between the receiver and this valve to 0.2 psia. Pressure with 74 to 15 psig. Evacuate and pressure two more times. Then shut the sampling line valve nearest the receiver to keep a blanket of 74 in this section of pipe and disconnect the receiver.
- (6) When the contents are melted, the hot water flow through the furnace can be reduced to avoid excessive cylinder pressures while waiting to drain cylinder.
- (7) Before draining cylinder check the pressure (PI-174) and level (PI-178) in blowcase F-136 to see that F-136 is ready to receive the charge. To drain the cylinder, open the header valve in line PL-51.
- (8) When the cylinder is empty, close the header valve in line

PI-51 and open header valve in line RV-52, venting the cylinder and piping connections. The cylinder is empty when the reading of PI-454 does not depend on which RV-53 valve is open, with the instrument and RV-53 pipe full of liquid.

- (9) Shut off the hot water flow to furnace.
- (10) With the header valve in PI-51, closed, purge the cylinder and piping of 616 as follows:

Pressure with 74 to 15 psig and vent through RV-51 ten times. Then evacuate to 0.2 psia through EV-16. Pressure again to 15 psig and re-evacuate to as low a pressure as possible.
- (11) After purging, close the cylinder valves and the last valves in both PI-51 and RV-52 before disconnecting the piping from the cylinder so as to maintain drums and as much piping as possible in a purged condition.
- (12) Break the piping connections and remove the cylinder from the furnace.

(c) Blowcase F-136

Blowcase F-136 is a means of transferring liquid 616 in the absence of a pump capable of doing the job, from the cylinders unloaded in waste furnaces E-131, to the tower feed tanks F-131. Located in the basement of the building it is charged by gravity from the liquid waste shipping cylinders. When filled, the contents are blown by high pressure 74 to either tower feed hold-up tank F-131A or B, located on the second floor. A sample connection is provided on each drum.

FUNCTION

The pressure controller is used when charging the blowcase from the cylinders to prevent pressure buildup. It controls the action of vent valve CV-316. For this service it is set to maintain a pressure of 20 psig. After charging the blowcase and before blowing the contents to the feed tanks, place CV-316 on manual operation and close it.

(1) Filling

NORMAL
OPERATION

With the drum vented of 74 and the pressure controller set to maintain a maximum drum pressure of 18 to 20 psig, it is charged by gravity from waste cylinders through header PL-1 and line PL-2. Refer to Figure V.2-2.

Normal charge to the blowcase is the contents of two waste cylinders (8000 to 10,000 lbs of 616).

(2) Blowing

- (a) After charging, sample the contents. If the analysis shows that distillation is required the contents are transferred to the tower feed hold-up tanks through lines PL-2 and PL-3; if distillation is not required, they are blown directly to the vaporizers through lines PL-2, PL-3, PL-18 and PL-15.

(b) Position the valves as follows:

Place CV-316 on manual operation and close it. Close the double block valves in PL-1 at the junction of PL-1, PL-2 and PL-3. If the transfer is to be to the tower feed tanks, close the double block valves in PL-18 and the block valve in PL-41 at the junction of PL-3 and PL-41. If the transfer is to be to the vaporizers, close the block valve in PL-3 at the junction of PL-3 and PL-41. Open the block valve in the PL line at the tower feed tank or vaporizer to which the transfer is to be made. If the transfer is to be a feed tank, open the block valve in PL-3 at junction of PL-3 and PL-41. If the transfer is to be to the vaporizers, open the double block valves in PL-18. Leave the block valve in PL-2 at the blowcase closed.

- (c) When ready to blow, pressure the blowcase with 100 psig 74 through DN-30. Control the rate of transfer by manual control of the block valve in PL-2 at F-136. Watch the progress of blowing by use of the following instruments on auxiliary board "B".

PI-376, liquid level in F-136, to gauge rate of transfer.

PI-374 and PI-375 liquid levels in F-136A and B, to gauge rate of transfer.

PIC-315, pressure in blowcase.

PI-357, available 74 pressure.

- (d) As the liquid level in the blowcase nears the bottom, the transfer rate should be reduced and finally stopped when within 3" of bottom. Care must be taken to maintain a liquid seal in the blowcase to prevent blowing 74 into the feed tanks or vaporizers.

- (e) Close the double block valves in DN-30.

(3) Venting

To ready the blowcase for the next charge, vent the tank through RV-28. Place CV-316 back on automatic operation

with PIC-315 set to maintain 18 to 20 psig.

(4) Blowcase Leak

EMERGENCY OPERATIONS

If because of a leak or for any other reason the blowcase must be emptied of process liquid, take the following steps:

- (a) If, at the time the leak is discovered, the blowcase is being charged from the waste cylinder, shut off the supply by closing the double block valves in PL-1 at the junction of PL-1, PL-2 and PL-3.
- (b) Since the only way of transferring liquid from the blowcase is by blowing to the tower feed tank, even though the high pressures will aggravate the leak, proceed to transfer the contents to the feed tanks by blowing, as described above. Omit the sampling and have the block valve in PL-2 at the blowcase wide open. For these conditions control the transfer rate by manually throttling the 74 flow to tank.
- (c) When the tank has been emptied as much as possible (PL-2 extends to within 1-1/2" of the bottom), shut off the 74 and vent the blowcase by opening wide the valve in RV-28 and CV-316 (on manual operation).
- (e) When the residual liquid has all been vented (check by closing vent valves and watching pressure) purge blowcase of 616. Refer to Section V.2-n.
- (f) Purge the casing. Refer to Section III-6.
- (g) Shut off the casing electric heaters.

Shut down of the blowcase means that the unloading of waste cylinders must be stopped. This merely involves controlling the hot water supply to the waste furnaces so as to maintain the cylinder contents liquid without building up excessive cylinder pressures.

If sufficient inventory is in the tower feed hold-up tanks, the shut down of the blow case will not require the shut down of still E-131.

(5) Instrument Failures

(a) Pressure Instruments

If, because of failure, the pressure transmitter must be removed from service, isolate it by closing the valve in the pressure line; do not drain the blowcase. Purge the transmitter of 616 by use of line PU-35. It is

advised that the blowcase not be used for transferring 616 to the tower feed tanks while PEM-175 is out of service. However, if conditions warrant, it can be done. Such a condition would exist if because of depletion of the tower feed tank inventory, the waste cylinders must be unloaded to avoid shutting down tower E-131.

(b) Temperature Instruments

Failure of the thermocouple circuit does not necessitate taking the blowcase out of service. Pressure readings, taking into account non-condensibles possibly present, give some indication of the temperature.

(c) Level Instruments

Failure of the liquid level transmitter does not, unless it involves removal of the float or otherwise breaking the instrument's seal against process, require emptying tank of 616. It is advised, however, that if possible, the blowcase not be used to transfer 616 when the transmitter is damaged.

(d) Tower Feed Hold-up Tanks F-131A and B

Tower feed hold-up tanks, F-131A and B, serves as in-process storage for contaminated 616, smoothing out variations in the composition and supply of 616. Normally, they are charged by transfer, through line PL-3, of 616 from the blowcase, F-136. Any material in dumping drum F-135 can also be blown through line PL-41 to the feed tanks reprocessing. See Sections V.2-c and V.2-i respectively for these operations.

FUNCTION

Each feed tank has a total capacity of 108,200 lbs of 616 at 165° F (481.5 cubic feet) and when filled to a depth of 48", contains 84,300 lbs. This latter figure corresponds to approximately 11 days design operation of tower E-131 (7632 lbs per 24 hr day).

- (1) Still pot F-130 is gravity charged from the feed tanks through line PL-4. Refer to Section V.2-e for details of this operation.

NORMAL
OPERATION

A sample connection is provided in each feed tank.

- (2) Feed Tank Leak

EMERGENCY
OPERATION

If because of a leak or for any other reason a feed tank must be emptied of process liquid, take the following steps:

- (a) Open the valve in PL-33A (F-131A) or PL-33B (F-131B)

to drain the tank to the dumping drum. It is assumed that the valve in PL-31 at the junction of PL-30, PL-31 and PL-41, and the valve in PL-30 at the drum are open, as they normally should be.

- (b) If the feed tank is being charged from any source at the time the leak is discovered, shut off this supply.
- (c) When the tank has drained, close the drain valve in line PL-33A or PL-33B. Vent the tank through the safety valve by-pass RV-14A or RV-14B.
- (d) Purge tank of 616. Refer to Section V.2-n.
- (e) Purge casing. Refer to Section III.6.
- (f) Shut off the casing electric heaters.

The shutdown of one feed tank need not seriously interfere with the operation of the feed purification system because both the charging of the feed tanks and the charging of the still pot are intermittent operations. Thus by staggering these operations the most serious consequences of shutting down one tank is a reduction in 616 inventory of the plant.

(3) Instrument Failures

(a) Pressure Instruments

If, because of failure, a pressure transmitter must be taken out of service, there is no need to drain the feed tank. Isolate the transmitter from the tank by closing the valve in the pressure line. Purge the transmitter of 616 by use of line PU-20A or PU-20B. It is advised that the feed tank be placed in standby service; that is, do not use the affected tank to receive additional 616 or to charge the still pot. In standby condition the tank is protected against excessive pressures by the safety valve and the casing heater controls.

If the unaffected feed tank is not being charged or discharged, the pressures in the two tanks can be equalized by interconnecting the tanks through PL-3. Thus the undamaged pressure transmitter will serve both tanks.

(b) Temperature Instruments

Failure of the thermocouple circuit does not necessitate taking the drum offstream. Pressure readings will give the operator an indication of the temperature.

(c) Level Instruments

Failure of the liquid level transmitter does not, unless it involves removal of the float or otherwise breaking

instrument's seal against process, require draining of the feed tank. It is advised, however, that the feed tank be placed in standby service. If conditions warrant continued use of the affected feed tank, the operator using the last recorded liquid level, can gauge the tank contents by carefully recording the amounts charged to and withdrawn from the feed tank.

(e) Batch Still E-131 and Auxiliaries

The batch still E-131 and its auxiliary equipment (still pot F-130), condenser C-131 and reflux drum F-138) perform the actual purification.

FUNCTION

Still pot F-130 is gravity charged from tower feed hold-up drums F-131A and B. HF, 816 and other impurities are removed from the 616 by equilibrium distillation through packed tower E-131. Cuts of 616 vapor, taken overhead from still E-131, are condensed in condenser C-131 and the condensate is dropped through a separator into reflux drum F-138. Reflux overflows from F-138 and returns to the top of the tower. The system operates on total reflux until equilibrium is reached and the desired 616 purity is obtained in reflux drum F-138. At this time F-138 is by-passed and the 616 cut is dropped to a run down drum F-132A, B or C. When making the final cut to remove remaining small amounts of 616 it may not be possible to obtain the required purity of 616 in reflux drum F-138. In this event, the cut is dropped to reflux hold-up drum F-137 and held there until it can be returned to still pot F-130 for reworking with a subsequent batch. Refer to Figure V.2-2.

(1) Valve Positions

NORMAL
OPERATION

After charging the still pot, make certain that all the block valves on lines connected to the still pot are closed, except CV-361. The valve in PL-6 below the separator should be closed, as should the valve in PL-37 below the trap. The valves in PL-15, PL-35, PL-16 and PL-12 at the bottom of F-138 should be closed. The valve in PL-7 is open, as well the valve in RV-5. CV-362 is open. The amount of material taken in a cut is decided upon beforehand, basing the judgment on purity of the material in the batch, purity of desired product, etc., and the appropriate valve in either PL-8, PL-9, PL-10 or PL-11 is closed. The valves above are open and those below are closed. Figure V.2-7 is a calculated graph showing F-138 capacities corresponding to PL-377 readings and "step ladder" valve positions. When all these valves are positioned, heat is applied to F-130 and the overhead vapors are condensed in C-131 and collected in F-138. When the reflux drum is filled to the proper level the vaporization is continued at total reflux until the desired

purity is obtained in F-138, as determined by frequent analysis. When enough experience has been gained, and when it has been demonstrated that the instruments are reliable, this point may be determined by temperature and pressure measurements. Otherwise sampling will have to be used. At this point the valve in PL-7 is closed, that in PL-6 opened. The "step ladder" valves may then be closed. The contents of F-138 may then be dropped to one of the rundown drums through PL-16. Another cut may be taken if there is sufficient material in the still pot to give a cut of acceptable purity.

(2) 74 Top Purge

CV-440, the needle valve on Panel 4 of the main control board, is set to maintain as small a flow of 74 to F-138 as will give good operation. Too great a flow will sweep away too much gaseous 616 and thereby reduce operating efficiency. Once a good setting is found, XX-365 can be used to stop the 74 without disturbing the valve position.

The boilup rate in F-130 should be as high as possible without flooding the tower.

(3) Still Pot or Still Leak

EMERGENCY
OPERATION

If, because of a leak, or for any other reason the still pot or still must be emptied of process liquid, take the following steps:

- (a) Turn off the power to the still pot heaters.
- (b) Drain the still pot to transfer tank F-135. Block valve CV-360, which is located in the still area, is remotely operated by XX-363 from the Main Control Board.
- (c) Close the valve in PL-6 and any that are open in overflow lines at reflux drum. Open the valve in PL-7 and close the valve in RV-5.
- (d) When the still pot has drained, close the valve in PL-7 and valve CV-360. Vent the system through the safety valve by-pass RV-10 and through CV-170.
- (e) Purge the system of 616 (see Section V.2-7). The motor valve in line PU-44 is remotely operated from auxiliary board "C".
- (f) Shut off the casing electric heaters.
- (g) Do not enter the tower area until the ventilating system has removed all 616 vapors.

(4) Reflux Drum Leak

If because of a leak or for any other reason the reflux drum must be drained, take the following steps:

- (a) If refluxing through the reflux drum is in process, open the valve in PL-6 and close the valves in PL-7, RV-5, and in the overflow lines.
- (b) Open the valve in PL-12 to drain the reflux drum back into the still.
- (c) Reduce the heat input to the still pot to the minimum required to keep the contents liquid.
- (d) When the reflux drum has drained, close the valve in PL-12. Vent the drum through safety valve by-pass RV-7.
- (e) Purge the drum of 616. Refer to Section V.2-n.
- (f) Purge the casing. Refer to Section III.6.
- (g) Shut off the casing electric heaters.

(5) Instrument Failures

(a) Pressure Instruments

In case of failure of any one of the pressure transmitters in the system there should be no difficulty in continuing operation on total reflux provided no attempt is made to change the operating conditions. Isolate the transmitter from process by closing the block valve in the pressure line, which in the case of PBM-200 is valve CV-361, remotely operated by XI-364 from the Main Control Board.

If the pressure recording controller PRC-163 fails the still pressure is maintained by manually controlling CV-170 or by the use of the block valve in RV-7.

(b) Temperature Instruments

Failure of the thermocouples does not require discontinuing operation of the system on total reflux provided no attempt is made to change the operation conditions. This is true of thermocouple TE-199 if the heat input is lowered to avoid the possibility of overheating the tube surface. See Section III.3-d.

- (c) In case of the failure of the level instruments on the still pot, operation of the system on total reflux is

possible. If the source of the trouble is not external to the still pot, then the system must be shut down and emptied. Refer to Section V.2-e.3.

On failure of LBM-171 at the reflux drum, the operation can proceed using the fixed overflow levels until it is desired to take the drum out of service. Refer to Section V.2-e.1.

(f) Reflux Hold-up Drum F-137

Reflux hold-up drum F-137 serves as a receiver for the last cuts taken overhead from still E-131 until they can be recharged to still pot F-130 for reworking. It is gravity charged from reflux drum F-138 through PL-16, and is drained to the still pot through PL-17 and PL-4.

FUNCTION

Reflux hold-up drum has a total capacity, including external float chamber and piping of 325 lbs of 616 at 165°F (scale reading 100). At a liquid level of 1'-6" (scale reading 60) the vessel contains 210 lbs of 616 at 165°F.

If the required 616 purity cannot be obtained by reflux drum F-138 when taking a final cut on waste, the contents of the reflux drum are dumped through PL-16 to the reflux hold-up drum. When the still pot F-130 is being charged with a fresh batch of waste the material in the reflux hold-up drum is returned through PL-17 and PL-4 to the still pot.

NORMAL
OPERATION

(1) Reflux Hold-up Drum Leak

EMERGENCY
OPERATIONS

If because of a leak or for any other reason the drum must be drained, take the following steps:

- (a) If possible, dump contents to the still pot. Otherwise drain the contents through line PL-35 to the dumping drum. It is assumed that the valve in PL-31 at the junction of PL-30, PL-31 and PL-41 and the valve in PL-30 at the dumping drum are open, as they normally should be.
- (b) If F-137, at the time the leak is discovered, is being charged from F-138, shut off the supply.
- (c) When the drum has drained, close the drain valve in PL-35 and vent the drum through RV-19.
- (d) Purge the drum of 616. Refer to Section V.2-n.
- (e) Purge the casing. Refer to Section III.6.
- (f) Shut off the casing electric heaters.

(2) Instrument Failures

(a) Pressure Instruments

To remove PEM-196 from service, close the block valve in the pressure line to the transmitter. Purge the transmitter of 616 by use of FU-49.

(b) Temperature Instruments

In case of failure of the thermocouple circuit, pressure readings give some indication of temperature.

(c) Level Instruments

If the failure of LBM-195 involves removal of the float or otherwise breaking of the instrument's seal against process, drum F-137 must be removed from service, drained and purged.

(g) Run-down Drums F-132A, B and C

Overhead cuts from batch still E-131 are dropped by gravity from reflux drum F-138 through PL-13 to one of the three run-down drums (F-132A, B or C). After accumulating the cuts from a batch of waste, the receiver is sampled and, if the contents are up to specification, they are dropped into one of the vaporizers through PL-14 and PL-15. If unsatisfactory, the material is dropped into dumping drum F-135.

FUNCTION

Each drum has a total capacity of 13,000 pounds of 616 at 165°F (57.8 cubic feet) and when loaded with 6,720 pounds, corresponding to the overhead cuts from one batch of waste, it has a liquid level of 45" (scale reading 50).

(1) Transferred Pure Contents

NORMAL
OPERATION

When satisfactory purity of 616 has been attained in reflux drum F-138, it is drained to a run-down drum through PL-13. Collect in one drum all the overhead cuts of 616 taken on a single batch charge to still pot F-130. After accumulating these charges sample the drum's contents. If the analysis shows the material to be up to specification, drain the contents to whichever vaporizer is not on stream feeding Section 400. The normal operating pressure of the drums is 15 psig at 165°F.

(2) Transfer of Impure Contents

If the analysis shows the batch requires reworking, dump the contents to the dumping drum F-135 for subsequent transfer to the tower feed tanks. It is also possible by using PL-18 to dump a run-down drum to blowcase F-136 but this is not recommended because of

contamination of headers PL-15.

(3) Vessel Leakage

EMERGENCY
OPERATION

If because of a leak or for any other reason a run-down drum must be drained, take the following steps:

- (a) If contents are known to be pure, drain the material to an evaporator. If not, drain to dumping drum F-135. It is assumed that the valve in PL-31 at the junction of PL-30, PL-31 and PL-41, and valve in PL-30 at F-135 are open, as they normally should be.
- (b) When drained, close the drain valve and vent the vessel using safety valve by-pass RV-18A, B or C.
- (c) Purge the vessel of 616. Refer to Section V.2-n.
- (d) Purge the casing. Refer to Section III.6.
- (e) Shut off the casing electric heaters.

With one drum out of service, it may be necessary to collect the cuts from more than just one batch charge to F-130, before sampling the run-down drum in order to continue operating the still at normal rates and still have time to analyze samples.

(4) Instrument Failures

(a) Pressure Instruments

If a pressure transmitter must be taken out of service there is no need to drain the vessel. Isolate the transmitter by closing the valve in the pressure line. Purge the instrument of 616 by using line FU-22A, B or C. It is recommended that the drum be placed in standby service during which time it is protected by the safety valve and by the casing heater controls.

(b) Temperature Instruments

Failure of a thermocouple circuit does not require taking the vessel out of service. Pressure readings given an indication of the temperature.

(c) Level Instruments

Failure of a level transmitter, if it involves removal of the float or otherwise breaking the instrument's seal against process, requires taking the drum out of service.

(h) Vaporizers F-133A and B

Vaporizers F-133A and B, serve as the feed source for Section 400 when the Fresh Feed Vaporization System is not in

FUNCTION

operation. Operation of these units is staggered, that is, one vaporizer is feeding Section 400 while the other is being charged from the run-down drums.

Each vessel has a total capacity of 108,200 lbs of 165°F (481.5 cubic feet) and when filled to a depth of 48" contains 84,300 lbs. This latter figure corresponds to approximately 14 days of operation when feeding 6000 lbs per day, 9 days at 9300 lbs per day and slightly under 7 days at the design rate of 12,600 lbs per day.

- (1) The vaporizer that is not onstream feeding Section 400 is charged batchwise from the run-down drums. The bottom process heaters are off.
- (2) After filling and just before placing the vessel onstream, sample the contents. If they are not up to specification, drain the contents to the dumping drum.
- (3) When ready to place onstream, turn on the process heaters with the pressure recording controller in operation.
- (4) Place the freshly charged vaporizer onstream by opening the valve in PG-3A or B. Then take the other offstream by closing the valve in PG-3A or B and shutting off its process heaters. The normal operating pressure of the vaporizers is 15 psig at 165°F.
- (5) Vaporizer Failure

NORMAL
OPERATION

To avoid interrupting feed to Section 400 in case of failure of an evaporator, place the other evaporation onstream immediately even though it is not fully charged or sampled. Proceed to prepare the Fresh Feed Vaporization System for operation (see Section V.1) so that feed will not be interrupted if the disabled vessel must be down for a long time.

EMERGENCY
OPERATIONS

(6) Vaporizer Leak

If because of a leak or for any other reason a vaporizer must be drained, take the following steps:

- (a) If the affected vaporizer is onstream feeding Section 400, immediately place the other vessel in operation. Then shut the valve in the vapor line of the affected evaporator and turn off the process heaters.
- (b) Drain the vessel to dumping drum F-135 through PL-39. It is assumed that the block valve in PL-31 at the junction of PL-30, PL-31 and PL-41, and the valve in PL-30 at transfer tank are open, as they normally should be.

(c) When drained, close the valve in PL-39A or PL-39B and then vent the tank through the safety valve by-pass RV-23A or RV-23B.

(d) Purge tank of 616. Refer to Section V.2-n.

(e) Purge casing. Refer to Section III.6.

(f) Shut off the casing electric heaters.

Shutdown of one tank should not seriously interfere with overall operation. Prepare to switch operations over to the Fresh Feed Vaporization System and while doing this use the undamaged vaporizer to feed Section 400.

(7) Instrument Failures

(a) Pressure Instruments

If, because of breakdown, a pressure transmitter must be taken out of service, there is no need to drain the vessel. Place the vaporizer in standby service (that is, neither charge nor feed from it) and isolate the transmitter from it by closing the valve in the pressure line. Purge the transmitter of 616 by use of PU-37A or PU-37B. Failure of the pressure transmitter necessitates shutting the vessel down since it is necessary to know vaporizer pressures in order to feed Section 400 properly.

(b) Temperature Instruments

Failure of the thermocouple circuit does not necessitate taking vaporizer off stream. Pressure readings give an indication of temperature.

(c) Level Instrument

Failure of the liquid level transmitter does not, unless it involves removal of the float or otherwise breaking the instrument's seal against process, require draining of the drum. If at all possible, however, feeding from the drum should be halted while the level instrument is out of order.

(1) Dumping Drum F-135

To dumping drum F-135, located in the basement, can be drained the contents of any 616 process vessel in the plant, with the exception of blowcase F-136 and still bottoms drum F-134. Liquid 616 can be blown with high pressure 74 from this drum back to tower feed tanks F-131A and B for reprocessing.

FUNCTION

- (1) Normal operation consists in holding this tank in readiness for emergencies in other equipment. The casing electric heaters should be on and the following valves positioned as indicated:

NORMAL
OPERATION

Valve in PL-30 at transfer tank, open.

Valve in PL-31 at junction of PL-30, PL-31 and PL-41, open.

Valve in PL-40, open.

Valve in PL-41, closed.

Valve in PL-21, closed.

CV-322, set to automatically maintain maximum pressure of 20 psig.

- (2) Dumping of Other Process Vessels

EMERGENCY
OPERATION

With F-135 in readiness the contents of other vessels (except F-134 and F-136) can in an emergency be dumped by opening the block valve in the drain line at the affected vessel.

- (3) Blowing out of F-135

To transfer contents of F-135 to the tower feed tanks, use the following procedures:

- (a) Position the valves as follows:

Place CV-322 on manual operation and close it. Close the valve in PL-31 at the junction of lines PL-30, PL-31 and PL-41.

Close the valve in PL-3 at the junction of PL-3 and PL-41.

Open the valve in the PL line at the feed tank to which the transfer is to be made.

Open the valve in PL-41 at junction of PL-3 and PL-41.

Leave the valve in PL-30 at the drum closed.

- (b) When ready to blow, pressure transfer tank with 100 psig 74 through DN-31. Control the rate of transfer by operating the valve in PL-30 at the vessel.

- (c) As the liquid level in the drum nears the bottom,

reduce the transfer rate and stop it when the level is 3" from the bottom. Care must be taken to maintain a liquid seal.

- (d) Close the double block valves in DW-31
 - (e) Vent the tank through RV-32. Return CV-322 to automatic operation.
 - (f) Position the valves as indicated in Section V.2-i-1.
- (4) Dumping Drum Leak

In case of a drum leak proceed as follows:

- (a) Since the only way of transferring liquid from the vessel is by blowing, even though the high pressure will aggravate the leak, proceed to transfer the contents by the procedure outlined in Section V.2-i.3. However, have the valve in PL-30 at the tank wide open and control the transfer rate by controlling the 74 flow.
- (b) When emptied as much as possible by blowing (line PL-30 extends to within 1-1/2" of bottom) shut off the 74 and vent the tank by opening the valve in RV-32 and placing CV-322 on automatic operation.
- (c) When the residual liquid has all been vented (check by closing the valves and watching the pressure) purge the tank of 615. Refer to Section V.2-n.
- (d) Purge the casing. See Section III.6.
- (e) Shut off the casing electric heaters.

(5) Instrument Failures

(a) Pressure Instruments

If the pressure transmitter must be removed from service, isolate it by closing the block valve in pressure line; do not drain the tank. Purge the transmitter using PU-40. It is advised that the drum not be used to return liquid to the tower feed tanks at this time, however, if conditions warrant, it can be done.

(b) Temperature Instruments

Failure of the thermocouple circuit does not necessitate taking the drum out of service. Pressure readings, taken into account non-condensibles possibly present, give some indication of temperature.

(c) Level Instruments

Failure of the level transmitter does not, unless it involves removal of float or otherwise breaking the instrument's seal against process, require emptying the dumping drum. It is advised, however, with the level instrument out of order that liquid not be transferred from the drum.

If at the time of instrument failure the drum is filled to its normal level (3") any of the vessels in the building may be dumped into it without fear of over filling.

(j) Still Bottoms Drum F-134 and Filter G-131

Any 816 separated from the waste distilled in still E-131 FUNCTION remains in the still pot and is then run through filter G-131 to still bottoms drum F-134. The 816 collected in this drum is then blown by high pressure 74 to extractor F-144. It is also possible to collect bottoms from several batches in F-134, blow them directly to F-130, and re-distill for more complete recovery of 616.

The still bottoms drum has a liquid level of 30" when charged with 3900 lbs of 816 at 275°F (which corresponds approximately to bottoms accumulated from three batch distillations), or 12-1/2" when charged with 1300 lbs of 816 at 275°F. After cooling to 100°F, 1300 lbs of 816 will give a level of approximately 11-1/2".

(1) Filling

NORMAL
OPERATION

With the pressure controller set to maintain a maximum pressure of 20 psig and the cooling water temperature controller set for 100°F, the still bottoms drum is charged from the still pot through lines PL-20, PL-21 and PL-22 and filter G-131. Have the valves in PL-40, PL-23 and PL-24 closed. Control the flow rate by the valve in PL-21 at the junction of PL-21, PL-22 and PL-23. The cooling water should be flowing and the casing heaters are off, as they always should be. They have only been provided for very special circumstances.

(2) Blowing

- (a) When ready to transfer the still bottoms drum's contents to extractor F-144, place CV-324 on manual control and close it.
- (b) Close the valve in PL-21 at the junction of PL-21, PL-22 and PL-23 and the valve CV-360 (XX-363) at the still pot. Open the valve in PL-40 so that still pot

may be easily dumped in an emergency to transfer tank F-135.

(c) Open the valve in PL-23 at the junction of PL-21, PL-22 and PL-23 and the valve at the extractor F-144.

(d) Control the blowing rate by manually controlling the 74 pressure put on the drum by line CN-32. Use as low a pressure as possible.

(e) As the liquid level near the bottom, reduce the transfer rate and stop when the level is 3" from the bottom.

(3) Venting

Vent drum through line RV-36, return CV-324 to automatic operation.

(4) Dumping Drum F-135 out of Service

EMERGENCY
OPERATION

If in an emergency a batch of 616 must be removed from still pot F-130 and the dumping drum F-135 is not available, the still bottoms drum may be used. Later the material in F-134 may be blown back to F-130 through PL-21, PL-24, and PL-20. The casing heaters are then used to liquefy the 616.

(5) Still Bottoms Drum Leak

If a leak should develop in the still bottoms drum transfer by blowing (see Section V.2-j.2) to extractor F-144, as much of the liquid as possible (PL-22 extends to within 1/2" of the bottom. Vent the remainder through RV-34 and through CV-324 (on manual operation). The heaters may be used to facilitate venting.

Then purge the vessel (see Section V.2-n) and casing (see Section III.6).

(6) Instrument Failures

(a) Pressure Instruments

If the pressure transmitter must be removed from service, isolate it by closing the block valve in the pressure line; do not drain the tank. Purge the transmitter using PU-43. It is advised that the still bottoms drum not be used to transfer liquid to extractor at this time. However, if conditions warrant, it can be done.

(b) Temperature Instrument

If temperature indications are available though CV-431 is inoperative, the cooling water flow is controlled manually by the block valve in the CV-431 by-pass.

(c) Level Instrument

Failure of the level transmitter does not, unless it involves removal of the float or otherwise breaking the instrument's seal against process, require emptying the still bottoms drum. It is advised, however, that drum not be used to transfer liquid.

(k) 616 Absorption System

When the concentration of T in the water in one of the circulating tanks is built up to the desired level of 1.26 pounds of 616 per gallon of water, the other pump and tank are put on stream supplying the tower, and the enriched solution from the first tank is then pump out of the circulating system to the 616 recovery system.

DESCRIPTION
OF OPERATION

The circulating tanks each have a total capacity of 610 gallons of water and when filled to a depth of 31" contain 500 gallons. A charge of 500 gallons can absorb 630 lbs of 616 which represents a 7-1/2 minute "blow" at a rate of 1.4 pounds per second.

(1) Operating Instructions

- (a) Fill each circulating tank with 500 gallons of water.
- (b) Open the valves in AC-1A and AC-3A to flood the suction and discharge of pump J-133A.
- (c) Start pump J-133A. It is very important that pumps never be run dry.
- (d) Throttle the valve in AC-3A to place discharge head of 43 psi on the pump. Check the pump operation and if any unusual conditions are apparent, stop the pump and determine the cause. Refer to the manufacturer's literature.
- (e) With all valves listed below open, slowly open the valve in AC-4A at pump discharge:

The valves in AC-5A and AC-5B at the towers.

The valves in AC-9A upstream and downstream of the junction of AC-9A and AC-10.

The valve in AC-9B upstream of the junction of AC-9A and AC-10.

The valve in AC-10.

- (f) Adjust the valve in AC-3A to give 30 psi pressure at the spray nozzles.
- (g) The absorption system is now ready to receive vent gases from Building K-131.

(2) Normal Operation

- (a) Normal operation consists essentially in maintaining constant flow to the spray nozzles and taking periodic samples to determine condition of the solution.
- (b) When the concentration of T in the solution has built up to 1.25 pounds of 616 per gallon of water, start pump J-133B in operation using tank F-141B. Refer to Section V.2-k.1.
- (c) To shift pumps and tanks proceed as follows:
 - (1) Open the valve in AC-9B downstream of the junction of AC-9B and AC-10.
 - (2) Close the valve in AC-9A downstream of the junction of AC-9A and AC-10.
 - (3) Open the valve in AC-4B.
 - (4) Open the valve in AC-3A.
 - (5) Close the valve in AC-4A.
- (d) Pump J-133A can now be stopped.
- (e) Before pumping the exhausted solution in tank F-141A to precipitator F-142, refer to Section V.2-l.

(3) Emergency Operations

If the circulating tank should spring a leak, immediately swing over to the other tank and pump, and, if possible, start transferring contents of the leaky tank to precipitator F-142.

Wearing the proper protective clothing, wash the floor and any equipment on which the acid leaked down the floor drains to the sump. To dispose of the material accumulated in the sump, it is pumped back to a circulating tank or directly to precipitator F-142, using the special suction pipe.

(1) 616 Recovery System

Before pumping the acid solution from K-132 to the precipitator, the precipitator F-142 is charged with 60 gallons of 10% caustic solution at 203°F for every 100 pounds of 616 in the acid solution to be precipitated. Air is blown through the caustic solution to provide agitation and the acid solution is pumped in. Open steam is used as required to maintain a temperature of 203°F during the precipitation. After the acid solution from the 616 absorption system has been added to the caustic already in the precipitator, additional 10% caustic is added slowly to bring the pH to 10.0 to 10.5, at which point the precipitation of T as C-100 should be complete. The slurry is further agitated by circulating through slurry pump J-134 while adding the caustic.

DESCRIPTION
OF OPERATION

When the precipitation is complete, the slurry is pumped through filter press G-133. The cake is then washed free of fluoride with warm water made up in the precipitator. The filtrate and wash water is pumped to tank trucks (two 1500 gallon trucks are provided) which transport the liquor to Building K-1407 for disposal. The cake is dumped from the press and charged to barrels for storage.

OPERATING
INSTRUCTIONS

- (1) The 50% caustic is transferred to F-143 from the shipping drums in which it is purchased by blowing with air. The 50% caustic is available commercially in 55 gallon drums which are approximately 22" ID by 35" high and contain approximately 700 lbs of 50% caustic. The drums normally have a screwed connection (1-1/2") on the side and an off-center bung hole in one end.
- (2) Air pressure is available by tying into the 1" plant air line dropping from line A-1-2" (elevation 804'-0") to the hose connections at elevations 792'-0" and 777'-0" on column F-32. With the shipping drum located on the 795'-8" platform and as close as practical to F-143, an air pressure of 3.5 to 4.0 psig should be sufficient for transferring the caustic.
- (3) A normal charge of acid solution to the precipitator F-142 consists of 500 gallons of water which has absorbed 630 lbs of 616. This requires theoretically 5000 lbs of 10% caustic solution to precipitate the T compounds and neutralize the acidity. It is recommended that a 10% excess of 10% caustic solution be used.
- (4) To make up the 10% caustic solution in F-143, the following procedure is recommended:
 - (a) Charge approximately 390 gallons of water to F-143 which will result in a depth of 2'-6" on the straight portion of the shell.

(b) With the caustic make up tank's agitator in operation, add 1100 lbs of 50% caustic by blowing as follows:

- (1) With the valves in the caustic line between the shipping drum and F-143, and the 1/2" air vent valve open, start pressuring the caustic shipping drum by partially opening the 1/4" air supply valve. The caustic flow can be started by slowly closing the vent valve until the air pressure gauge reads 3.5 to 4.0 lbs.
- (2) When the desired volume of caustic has been transferred, close the caustic valve and immediately close the 1/4" air supply valve and release the drum pressure through the 1/2" vent valve.
- (3) After each blowing and particularly before disconnecting the caustic line from the shipping drum and F-143, drain the caustic line by opening the valves. When breaking the caustic line have a drain pail under the opening.
- (4) By controlling the cooling water flow through the jacket of F-143 and by controlling the caustic blowing rate, the temperature in F-143 can be controlled.
- (5) After adding the 50% caustic to F-143, the liquid level should be approximately 3'-1" on the straight.
- (6) Add sufficient additional water to F-143 to bring the caustic concentration to 10%, which should require approximately an additional 143 gallons of water. The level now is approximately 4'-4" on the straight.
- (7) The length of time the 10% caustic stands before using, the rate at which dilution was made, and the cooling water rate during dilution, determined whether steam will be needed in the jacket to heat the 10% caustic to about 203°F before using the precipitator.
- (8) Add to the precipitator 60 gallons of 10% caustic for every 100 lbs of 616 that is present in the acid solution to be treated.
- (9) When ready to add the acid solution, start agitation of the precipitator's contents with air. As the 500 gallons of acid solution is slowly pumped in from the circulating tanks in the 616 absorption system it may be necessary to use live steam for the agitation of F-142 in order to maintain a temperature of 203°F.

- (10) With the acid solution completely added, use slurry pump J-134 to agitate the batch by circulating the slurry mixture through line AC-15, by-passing the filter press.
- (11) While circulating the mixture, slowly add additional 10% caustic to bring the mixture's pH to 10.0 to 10.5 at which time precipitation should be complete.
- (12) Proceed to filter the mixture.
- (13) Extreme caution should be observed in handling of the caustic soda particularly in the "blowing" from drums to F-143. Manufacturers of caustic generally recommend the wearing of cotton clothing; rubber, heavy canvas or moleskin gloves; wide brimmed hats; either low acid-proof rubber shoes or a canvas shoe with rubber soles and heels; and goggles properly adjusted to the contour of the face, care being taken that they are provided with ample protection such as wide vision and side shields properly ventilated. A scarf or kerchief around the neck provides further protection.
- (14) To build-up an initial cake on the cloths of filter press G-133, start the flow through the press and return the filtrate to precipitator through line AC-15.
- (15) When sufficient cake has been built up to insure a clear filtrate, turn the filtrate flow out to the tank truck.
- (16) After completion of the filtration prepare, in the precipitator, a batch of warm wash water, using live steam.
- (17) Wash the filter cake until it is free of fluorides, taking the wash water to the tank trucks.
- (18) When sufficiently washed, open the press and dump the cake into the pan provided under the press. The precipitate is then shoveled from the pan into drums for storage.

(m) 816 Extraction System

The stripped 816 from F-130 collected as still bottoms in still bottoms drum F-134 is blown to the 816 extraction system. In this system, traces of 616 are removed by agitation with a dilute potassium carbonate solution and consequently separation of the aqueous layer from the 816 layer. The 816 is then returned in

FUNCTION

drums to the main coolant system of the plant. Equipment of the 816 extraction system consists of:

- (a) Extractor F-144 in which the still bottoms are agitated with 2% K_2CO_3 solution.
 - (b) K_2CO_3 solution make up tank F-145 for the preparation of 2% carbonate solution.
 - (c) Separator E-133 in which the agitated mixture from F-144 is allowed to separate on standing.
 - (d) Separator pump J-136 to return the aqueous layer from E-133 to F-144 for reuse or to precipitator F-142 for recovery of T compounds.
- (1) Prepare a 2% solution of K_2CO_3 in F-145. A batch of solution equal in weight to the weight of 816 to be treated is required. OPERATION
 - (2) When ready to treat the still bottoms, run 80 gallons of 2% K_2CO_3 solution (approximately 675 lbs of 2% K_2CO_3 solution) into F-144. This will charge F-144 to a depth of 12" above the bottom tangent line.
 - (3) Transfer slowly to F-144 by blowing (see Section V.2-j.2) 44 gallons of bottoms (approximately 675 lbs of 816). This will fill F-144 an additional 10 inches.
 - (4) Agitate the mixture for 15 minutes.
 - (5) With the agitator running, draw a portion of the mixture from F-144 to the separator. An extractor batch will yield approximately four separator batches.
 - (6) Allow the mixture to stand in E-133 until it separates into two sharp layers.
 - (7) Open the bottom outlet valve on the separator and slowly run the bottom (816) layer into the 816 shipping drums. As the interface between two layers passes float of LBC-262, valve CV-289 closes.
 - (8) Pump the upper (K_2CO_3 solution) layer to precipitator F-142 or back to F-144 for reuse, using J-136.
 - (9) Shipping drums of washed 816 are transferred to the 816 drying system of the main plant for reuse.

(n) Purging Operation

Following exposure of equipment or piping to atmospheric MOISTURE PURGING

moisture, purging is necessary to reduce the dewpoint in the equipment to minus 40°F before exposure to 616. Moisture purging of the demountable cylinder connections in the shipping drum feed room and the liquid waste unloading room is regularly necessary. It is essential that the 616 vacuum pumps, J-132A and J-132B not be used for this service. Instead use the moisture purging pumps, J-138A and J-138B, and the moisture purging piping designated as WE.

The purging procedure is as follows:

- (1) Evacuate the system to 0.2 psia.
- (2) Pressure to at least 15 psig with 74.
- (3) Repeat steps (1) and (2).
- (4) Evacuate to as low a pressure as possible before placing system in 616 service. This final evacuation is to reduce to a minimum the non-condensable gases trapped in the system.

To prevent injury to operators or workmen, it is necessary, before opening for inspection or maintenance a system that has been exposed to 616, to reduce the 616 concentration to at least one part per million by volume. Before purging, drain the system of liquid 616 and vent the system until no residual liquid remains. If possible, maintain the casing heaters in operation while venting to hasten evaporation. Use of the pressure instruments will show the presence of liquid by an increase in pressure when the system is isolated. All process equipment is provided with a tie to the G-74 distribution system and to the 616 evacuation system, in most cases by means of PU lines.

616 PURGING

After draining and venting, purge of 616 is as follows:

- (1) Pressure to at least 15 psig with 74 and vent. Repeat this alternate pressuring and venting nine additional times.
- (2) Evacuate to 0.2 psia.
- (3) Pressure to at least 15 psig with 74.
- (4) Exhaust to 0.2 psia.
- (5) If the equipment is to standby for sometime, pressure to 15 psig with 74.

(o) Coolant System

- (1) Before starting up, the entire system must be thoroughly cleaned of grease, organic matter and moisture. As a final STARTUP

precaution, blow through with dry air.

- (2) Charge surge drum F-139 through lines CO-9 and CO-403 from Section 400.
- (3) Check carefully to see that the pump and its motor has been lubricated in accordance with manufacturer's recommendations.
- (4) Fill pump J-131A with 816 from the surge drum through line CO-1, equalizing the levels by keeping line CO-10 open. Keep all valves in the suction line and in the gas pressure equalizing line to the surge tank wide open.
- (5) Open the necessary valves to circulate through lines CO-2, CO-3, CO-6 and the by-pass around CV-297 back to the surge drum. With the discharge valve at the pump open a few turns start the pump. As soon as it is started, open the valve. Do not run the pump with the discharge valve closed for any long period of time, as the heat which is generated rapidly in the pump case will cause eventual damage.
- (6) The pump packing should be given careful attention during the startup period to prevent overheating. If the packing tends to heat rapidly, relieve the packing pressure by backing off the gland.
- (7) Throttle the valve in CO-6 to bring the discharge head on the pump to its normal value of 138 psi (40 gpm).
- (8) If any excessive vibration is observed, stop the pump and investigate. Refer to the manufacturer's literature.
- (9) Set pressure controller FIC-298 to maintain 82 psig upstream of CV-297. Close the by-pass and open the valve in CO-6 wide.
- (10) With FRC-159 on manual control, start the cooling water flow through cooler C-133 to maintain an exit temperature at the cooler of 155°F. If, as at initial startup, the 316 temperature is below 155°F, use the low pressure steam, available at cooler C-133, to preheat the coolant.
- (11) With the globe valve in CO-4 wide open and the valve in CO-5 at condenser C-131 open, close the valve in CO-6.
- (12) After sufficient time has elapsed to bleed all the trapped gas from the condenser, close the valve in CO-5.

- (13) The coolant system is now ready for turning 616 vapor into the condenser. Place CV-302 on automatic control.

During normal operations the following conditions should be maintained:

NORMAL
OPERATION

- (14) The coolant pressures at all points should be higher than either the 616 or water pressure at that point.
- (15) The coolant temperature at the exit of C-133 is 155°F.
- (16) The 616 temperature at the exit of C-131 is 165°F.

To close down:

SHUTDOWN

- (17) Shut off the power to the pump motor.
- (18) Close the valve at the pump's suction (line CO-1A)
- (19) To drain the bulk of the system into surge drum, open the following valves:
- (a) The valve in CO-5 at C-131.
 - (b) The globe valve in CO-4 at C-131.
 - (c) The valve in the by-pass around CV-297.
 - (d) The valve in CO-6.
 - (e) The valve in CO-2A at the pump discharge.
 - (f) The valve in CO-8A.
- (20) To blow the remaining 816 from the lower portion of the piping, first crack open the valve in DA-7 to blow out lines CO-2 and CO-2A. Close the valves in CO-2A and DA-7.
- (21) Blow out pump J-131A through CO-8A using the air pressure available through DA-2 and CO-11.

DISTRIBUTION

1. K-25 Site Records (RC)
2. ChemRisk/Shonka Research Associates
3. S. G. Thornton (K-25 EMD)
4. DOE Public Reading Room